END OF METHOD CONTROL PARAMETERS

Quantitation method
(b)
(7)(C) 8/11/14

Method Comments:

C:\MSDCHEM\1\METHODS\\(\bar{1} \) RP1581_051914.M Fri Aug 08 13:44:40 2014

Control Information _____

Sample Inlet : GC Injection Source : GC ALS Mass Spectrometer: Enabled

Oven

Equilibration Time 0.5 min Oven Program On 35 °C for 2 min

then 20 °C/min to 325 °C for 3 min

Run Time

```
Front Injector
                                            10 µL
Syringe Size
Injection Volume
                                             1 μL
Injection Repetitions
                                             2
Solvent A Washes (PreInj)
Solvent A Washes (PostInj)
                                              3
Solvent A Volume
                                             8 µL
                                              2
Solvent B Washes (PreInj)
Solvent B Washes (PostInj)
                                              3
Solvent B Volume
                                              8 µL
Sample Washes
                                              1
Sample Wash Volume
Sample Pumps
                                              8 µL
                                              3
Dwell Time (PreInj)
Dwell Time (PostInj)
                                             0 min
                                        0 min
300 µL/min
Solvent Wash Draw Speed
Solvent Wash Draw Speed 300 µL/min Solvent Wash Dispense Speed 6000 µL/min 300 µL/min
Sample Wash Draw Speed
                                          300 µL/min
                                      6000 μL/min
6000 μL/min
Sample Wash Dispense Speed
Injection Dispense Speed
                                             0 sec
Viscosity Delay
Sample Depth Disabled
```

Back Injector		
Syringe Size	10	μL
Injection Volume	1	$\mu \mathbf{L}$
Injection Repetitions	1	
Solvent A Washes (PreInj)	0	
Solvent A Washes (PostInj)	0	
Solvent A Volume	8	$\mu ext{L}$
Solvent B Washes (PreInj)	0	
Solvent B Washes (PostInj)	0	
Solvent B Volume	8	$\mu extsf{L}$
Sample Washes	0	
Sample Wash Volume	8	μL
Sample Pumps	6	
Dwell_Time (PreInj)	0	min
Dwell Time (PostInj)	0	min
Solvent Wash Draw Speed	300	μL/min
Solvent Wash Dispense Speed		μL/min
Sample Wash Draw Speed	300	μL/min
Sample Wash Dispense Speed		μL/min
Injection Dispense Speed	6000	µL/min
Viscosity Delay	0	sec
Sample Depth Disabled		

Front SS Inlet He Mode Heater Pressure Total Flow Septum Purge Flow Gas Saver Split Ratio Split Flow	Split On 275 °C On 16.494 psi On 114.93 mL/min On 3 mL/min On 30 mL/min After 2 min 20 :1 106.6 mL/min
Back SS Inlet He Mode Heater Pressure Total Flow Septum Purge Flow Gas Saver Split Ratio Split Flow	Split Off Off Off Off Off 100 :1 277.4 mL/min
Column #1	
450 °C: 25 m x 320 μm x 0 μm In: Front SS Inlet He Out: Front Detector TCD	
(Initial) Pressure Flow Average Velocity Holdup Time Flow Program 5.3298 mL/min for 0 min Run Time	35 °C 16.494 psi 5.3298 mL/min 70.102 cm/sec 0.59437 min Off 19.5 min
Column #2	
450 °C: 25 m x 320 μm x 0 μm In: Back SS Inlet He Out: Front Detector TCD	
(Initial) Pressure Flow Average Velocity Holdup Time Pressure Program 10 psi for 0 min Run Time	35 °C 10 psi 2.774 mL/min 43.399 cm/sec 0.96009 min Off 19.5 min
Front Detector TCD Heater Reference Flow Makeup Flow Const Col + Makeup Negative Polarity Filament	On 300 °C Off On 20 mL/min Off Off Off

Signals

Front Signal Test Plot Test Plot Test Plot

Save On Save On Save On Save On

MS ACQUISITION PARAMETERS

General Information

Tune File : dftppnew051614.u

Acquistion Mode : Scan

MS Information _____

Solvent Delay

: 3.50 min

EMV Mode

: Relative

Relative Voltage

: -71

[Scan Parameters]

Low Mass High Mass : 35.0

Threshold

: 500.0

Sample #

A/D Samples 8

Plot 2 low mass Plot 2 high mass

: 50 : 3 : 50.0 : 550.0

[MSZones]

MS Source MS Quad

: 230 C maximum 250 C

: 150 C maximum 200 C

END OF MS ACQUISITION PARAMETERS

TUNE PARAMETERS for SN: <offline>

Trace Ion Detection is OFF.

EMISSION ENERGY

34.600

70.000

REPELLER : IONFOCUS :

29.960

ENTRANCE_LE :

EMVOLTS

MASSOFFSET :

90.200

Actual EMV :

: 1200.000

GAIN FACTOR:

<Unable to calculate gain facto</pre>

r.>

AMUGAIN: 4095.000
AMUOFFSET: 255.000
FILAMENT FILAMENT : DCPOLARITY : 1.000 0.000 ENTLENSOFFS : 25.100 MASSGAIN :

-2048.000 -499.000

END OF TUNE PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS\(\begin{array}{c} \beg

Percent Report Settings

Sort By: Signal

Output Destination

Screen: No Printer: No File: No

-Integration Events: Meth Default

Generate Report During Run Method: Yes

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search Minimum Quality

C:\Database\NIST05a.L 0

Integration Events: Meth Default

Report Type: Summary

Output Destination

Screen: No Printer: No File: No

Generate Report During Run Method: Yes

Quantitative Report Settings

Report Type: Detailed

Output Destination Screen: No Printer: Yes

File: epatemp.txt

Generate Report During Run Method: No

Freedom Industies

Calibration Last Updated: Wed Aug 06 11:32:39 2014

Reference Window: 2.00 Minutes
Non-Reference Window: 1.00 Minutes

(P1581_051914.M Fri Aug 08 13:44:39 2014

```
Default Multiplier: 1.00
Default Sample Concentration: 0.00
Compound Information
                                                 ( )
     Cyclohexanol
Ret. Time 4.613 min., Extract & Integrate from 4.113 to 5.113 min.
                         Pct. Unc.(rel)
                                            Integration
             Rel Resp.
Signal
                                            *** METH DEFAULT ***
Tgt 57.10
                                            *** METH DEFAULT ***
                57.10
                             20.0
      82.10
Q1
                                            *** METH DEFAULT ***
                             20.0
                43.80
      67.10
Q2
Lvl ID
         Conc (ug/mL)
                       Response
            50.000
                         156539
            50.000
                         155813
                         169255
3
            50.000
                         157595
            50.000
4
            50.000
                         151352
5
            50.000
                          168333
6
7
            50.000
                          167964
            50.000
                          163415
Qualifier Peak Analysis ON
Curve Fit: Avg. RF
                                                 ( )
   MCHM (2 peaks combined)
Ret. Time 6.530 min., Extract & Integrate from 6.030 to 7.030 min.
Signal
                        Pct. Unc.(rel)
                                             Integration
             Rel Resp.
                                             *** METH DEFAULT ***
      55.10
Tgt
                                             *** METH DEFAULT ***
                39.40
                             20.0
      97.10
Q1
                                             *** METH DEFAULT ***
                30.30
                             20.0
      81.10
Q2
                        Response
Lvl ID
         Conc (ug/mL)
             0.470
                            874
                            2156
             0.946
2
                            7147
             2.840
3
             4.730
                           11471
4
             9.460
                          22258
5
                          166406
6
            47.300
                          367991
            94.600
7
           189.000
                          789691
Qualifier Peak Analysis ON
Curve Fit: Linear
 3) PPH
Ret. Time 7.784 min., Extract & Integrate from 7.284 to 8.284 min.
             Rel Resp. Pct. Unc. (rel)
                                             Integration
Signal
                                             *** METH DEFAULT ***
Tqt
      94.10
                                             *** METH DEFAULT ***
                             20.0
      77.10
                23.70
01
                                             *** METH DEFAULT ***
                18.60
                             20.0
     152.10
Q2
Lvl ID
         Conc (ug/mL)
                        Response
                             604
1
              0.470
              0.946
                            1506
2
                            6257
3
             2.840
             4.730
                           10474
             9.460
                           21573
5
                          189499
             47.300
6
             94.600
                          441478
                         1023617
            189.000
```

Correlation Window: 0.10 minutes

curve fit: Linear

4) 1,4-CHDM

()

Ret. Time 8.592 min., Extract & Integrate from 8.092 to 9.092 min.

```
Rel Resp. Pct. Unc. (rel)
Signal
                                           Integration
    95.10
                                           *** METH DEFAULT ***
Tgt
                                           *** METH DEFAULT ***
QĪ
     67.10
                41.40
                           20.0
     41.10
                53.20
                           20.0
                                           *** METH DEFAULT ***
Q2
```

Lvl II	Conc (ug/mL)	Response
$T \wedge T = T \Gamma$	cone (ug/mb)	Keaponae
1	0.470	280
2	0.946	545
3	2.840	2435
4	4.730	4360
5	9.460	8585
6	47.300	74923
7 .	94.600	188094
8	189.000	411739

Qualifier Peak Analysis ON

Curve Fit: Linear

5) DMCH-1,4-DC ()

Ret. Time 9.152 min., Extract & Integrate from 8.652 to 9.652 min.

```
Rel Resp.
Signal
                        Pct. Unc.(rel)
                                           Integration
Tgt 81.10
                                           *** METH DEFAULT ***
                                           *** METH DEFAULT ***
     108.10
                            20.0
Q1
                36.20
                                           *** METH DEFAULT ***
Q2
    140.10
                63.60
                            20.0
Lvl ID
         Conc (ug/mL)
                       Response
             0.470
                           646
             0.946
2
                           1398
3
             2.840
                          5114
                          7748
            4.730
             9.460
                         18343
            47.300
                         130770
7
            94.600
                         293609
```

Qualifier Peak Analysis ON

189.000

Curve Fit: Linear

6) di-PPH ()

639126

Ret. Time 9.856 min., Extract & Integrate from 9.356 to 10.356 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 59.10			*** METH DEFAULT ***
Q1 94.10	30.00	20.0	*** METH DEFAULT ***
Q2 210.20	9.00	20.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/mL)	Response
1		0.470	401
2		0.946	1017
3		2.840	2992
4		4.730	4799
5		9.460	11129
6		47.300	83676
7		94.600	214860
8		189.000	-1

Qualifier Peak Analysis ON Curve Fit: Linear



END OF DATA ANALYSIS PARAMETERS

Fri Aug 08 13:44:41 2014

Data Path : C:\msdchem\1\DATA\0 (7) \RP1581\

Data File: 051914_001.D

Acq On: 19 May 2014 11:42 am

Operator: (b) (7)

: DFTPP Sample Misc : DFTPP

ALS Vial : 1 Sample Multiplier: 1

Quant Time: Aug 06 11:31:39 2014

Quant Method: C:\msdchem\1\METHODS\60(7)RP1581_051914.M Quant Title: Freedom Industies QLast Update: Wed Aug 06 11:28:36 2014

Response via : Initial Calibration

Compound	R.T. QIon	Response	Conc Units Dev(Min)
System Monitoring Compounds 1) Cyclohexanol	0.000 57	0	0.00 ug/mL
Target Compounds 2) MCHM (2 peaks combined) 3) PPH 4) 1,4-CHDM 5) DMCH-1,4-DC 6) di-PPH	0.000 0.000 0.000 0.000 0.000	0 0 0 0	Qvalue N.D. N.D. N.D. N.D.

Data Path: C:\msdchem\1\DATA(b)(7)(C) RP1581\

Data File : 051914 001.D

Acq On : 19 May 2014 11:42 am

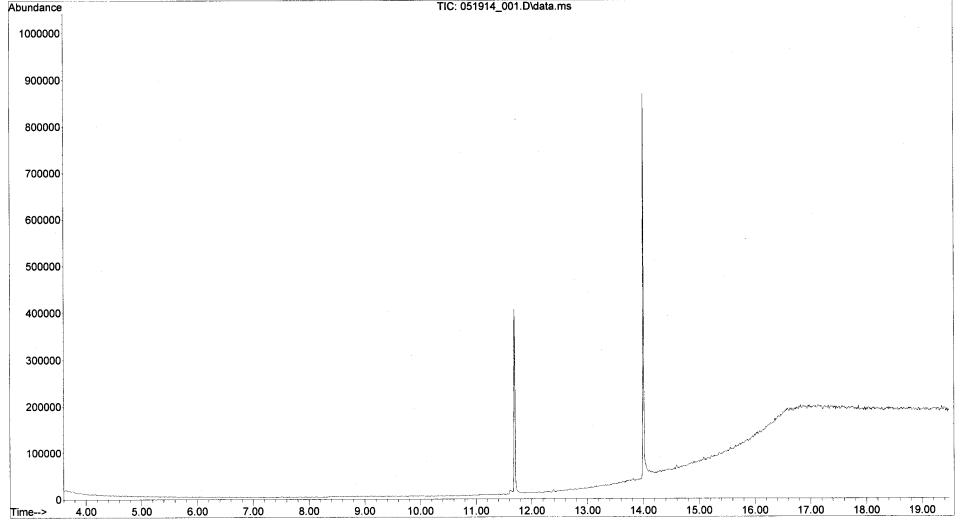
Operator : (b) (7)(C)
Sample : DITT
Misc : DFTPP

ALS Vial: 1 Sample Multiplier: 1

Quant Time: Aug 06 11:31:39 2014

Quant Method: C:\msdchem\1\METHODS(b)(7)(C).P1581_051914.M

Quant Title : Freedom Industies
QLast Update : Wed Aug 06 11:28:36 2014
Response via : Initial Calibration



Data Path : C:\msdchem\1\DATA(b)(7)(C) RP1581\

Data File: 051914_002.D
Acq On: 19 May 2014 12:17 pm
Operator: (b) (7)(C)

Sample : Blank Misc : Blank

: 2 ALS Vial Sample Multiplier: 1

Quant Time: Aug 06 11:32:06 2014

RP1581 051914.M Quant Method : C:\msdchem\1\METHODS

Quant Title : Freedom Industies QLast Update : Wed Aug 06 11:28:36 2014

Response via : Initial Calibration

Compound	R.T. QIon	Response	Conc Units Dev(Min)
System Monitoring Compounds 1) Cyclohexanol	0.000 57	0	0.00 ug/mL
Target Compounds 2) MCHM (2 peaks combined) 3) PPH 4) 1,4-CHDM 5) DMCH-1,4-DC 6) di-PPH	0.000 0.000 0.000 0.000	0 0 0 0	Qvalue N.D. N.D. N.D. N.D. N.D.

Data Path : C:\msdchem\1\DATA(b)(7)(C) RP1581\

Data File : 051914_002.D

Acq On : 19 May 2014 12:17 pm

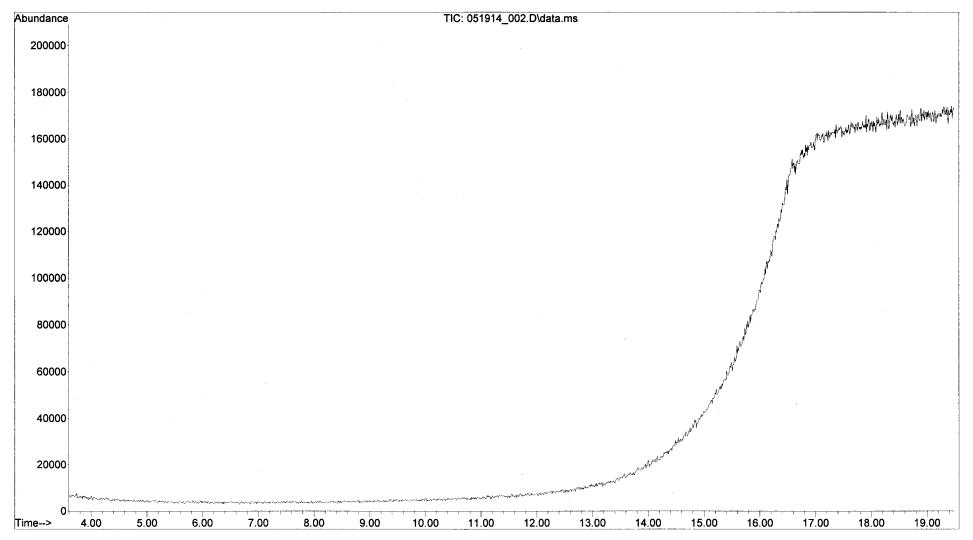
Operator : (0) (1)
Sample : (C) k
Misc : Blank

ALS Vial : 2 Sample Multiplier: 1

Quant Time: Aug 06 11:32:06 2014

Quant Method: C:\msdchem\1\METHODS RP1581_051914.M

Quant Title : Freedom Industies QLast Update : Wed Aug 06 11:28:36 2014



Data Path: C:\msdchem\1\DATA\(b)(7) \RP1581\

Data File : 051914_003.D

: 19 May 2014 12:55 pm : (b) (7)(C) Acq On

Operator

Sample ug/mL : 0.5

Misc : 0.5

ALS Vial : 3 Sample Multiplier: 1

Quant Time: Aug 06 11:40:07 2014

Quant Method: C:\msdchem\1\METHODS () (7) RP1581_051914.M Quant Title: Freedom Industies QLast Update: Wed Aug 06 11:32:39 2014

Compound	(b) (7)(C)		Response	Conc Ur	nits De	v(Min)
System Monitoring Compounds 1) Cyclohexanol	4.613	57	156539	48.53	ug/mL	0.00
Target Compounds 2) MCHM (2 peaks combined) 3) PPH 4) 1,4-CHDM 5) DMCH-1,4-DC 6) di-PPH	6.530 7.784 8.592 9.152 9.732	55 94 95 81 59	842m 548m 280m 646m 401m	4.27 4.08 3.18	ug/mL ug/mL ug/mL ug/mL ug/mL	value

^{(#) =} qualifier out of range (m) = manual integration (+) = signals summed

Data Path : C:\msdchem\1\DATA (b) (7)(C) RP1581\

Data File : 051914_003.D

Acq On : 19 May 2014 12:55 pm

Operator : (b) (7)(C)

Sample : 0.5 ug/mL

Misc : 0.5

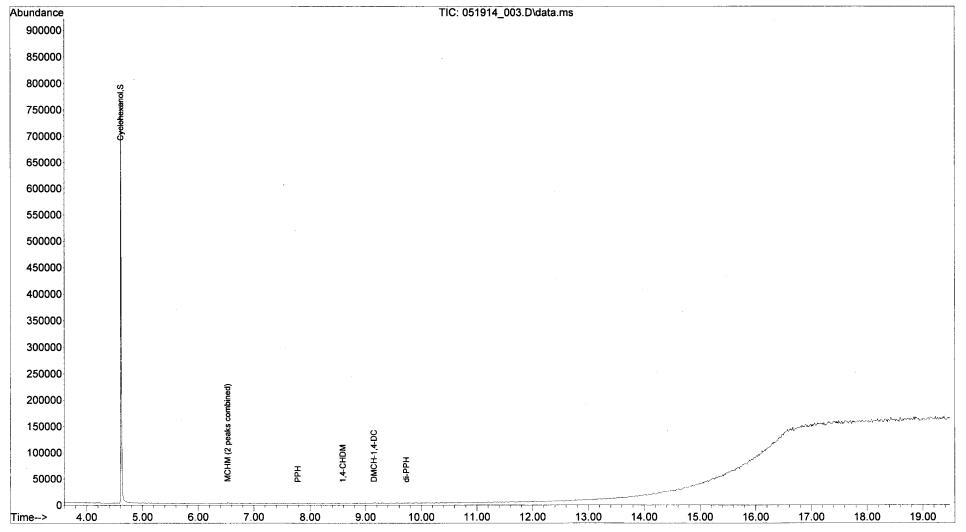
ALS Vial : 3 Sample Multiplier: 1

Quant Time: Aug 06 11:40:07 2014

Quant Method: C:\msdchem\1\METHODS(b)(7) RP1581_051914.M

Quant Title : Freedom Industies

QLast Update: Wed Aug 06 11:32:39 2014



Data Path : C:\msdchem\1\DATA\(\begin{picture}(0) (7) \\ (1) \end{picture} \RP1581\

Data File : 051914_004.D Acq On : 19 May 2014 1:28 pm

: (b) (7) : I ug/mL : 1 Operator Sample

Misc

: 4 ALS Vial Sample Multiplier: 1

Quant Time: Aug 06 11:41:21 2014

Quant Method: C:\msdchem\1\METHODS (0) (7) RP1581_051914.M

Quant Title : Freedom Industies QLast Update: Wed Aug 06 11:32:39 2014

Response via : Initial Calibration

Compound	(b) (7)(C)		Response	Conc Units I	Dev(Min)
System Monitoring Compounds 1) Cyclohexanol	4.613	57	15581 <u>3</u>	48.30 ug/ml	L 0.00
Target Compounds 2) MCHM (2 peaks combined) 3) PPH 4) 1,4-CHDM 5) DMCH-1,4-DC 6) di-PPH	6.530 7.784 8.582 9.152 9.846	55 94 95 81 59	2156m 1506m 545m 1398m 1017m	3.18 ug/ml 4.45 ug/ml 4.20 ug/ml 3.41 ug/ml 3.04 ug/ml	<u>.</u>

Data Path : C:\msdchem\1\DATA(b)(7)(C),RP1581\

Data File : 051914 004.D

: 1

Acq On : 19 May 2014 1:28 pm

Operator : (b) (7)(C)
Sample : I ug/mL

Misc

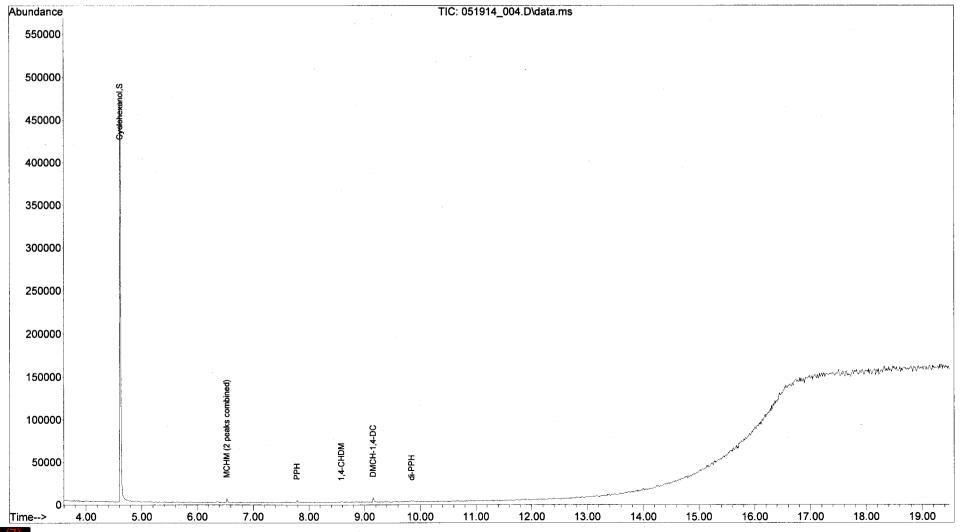
ALS Vial : 4 Sample Multiplier: 1

Quant Time: Aug 06 11:41:21 2014

Quant Method: C:\msdchem\1\METHODS (6) (7) RP1581 051914.M

Quant Title : Freedom Industies

QLast Update: Wed Aug 06 11:32:39 2014



Data Path : C:\msdchem\1\DATA (b) (7)(C) RP1581\

Data File : 051914_005.D

: 19 May 2014 Acq On 2:00 pm

Operator Sample

d/mL Misc 3 :

ALS Vial : 5 Sample Multiplier: 1

Quant Time: Aug 06 11:42:27 2014

Quant Method: C:\msdchem\1\METHODS
Quant Title: Freedom Industies
QLast Update: Wed Aug 06 11:32:39 2014
Response via: Initial Calibration RP1581 051914.M

Compound	(b) (7)(C)		Response	Conc Units I	Dev(Min)
System Monitoring Compounds 1) Cyclohexanol	4.623	57	169255	52.47 ug/mI	0.01
Target Compounds 2) MCHM (2 peaks combined) 3) PPH 4) 1,4-CHDM 5) DMCH-1,4-DC 6) di-PPH	6.530 7.774 8.582 9.152 9.846	55 94 95 81 59	7148m 6493m 2435m 5114m 3031m	4.37 ug/mI 5.38 ug/mI 5.07 ug/mI 4.51 ug/mI 3.94 ug/mI	, ,

Data Path : C:\msdchem\1\DATA\(0)(7)(C) RP1581\

Data File : 051914 005.D

Acq On : 19 May 2014 2:00 pm

Operator : (b) (7)
Sample : 3 ug/mL

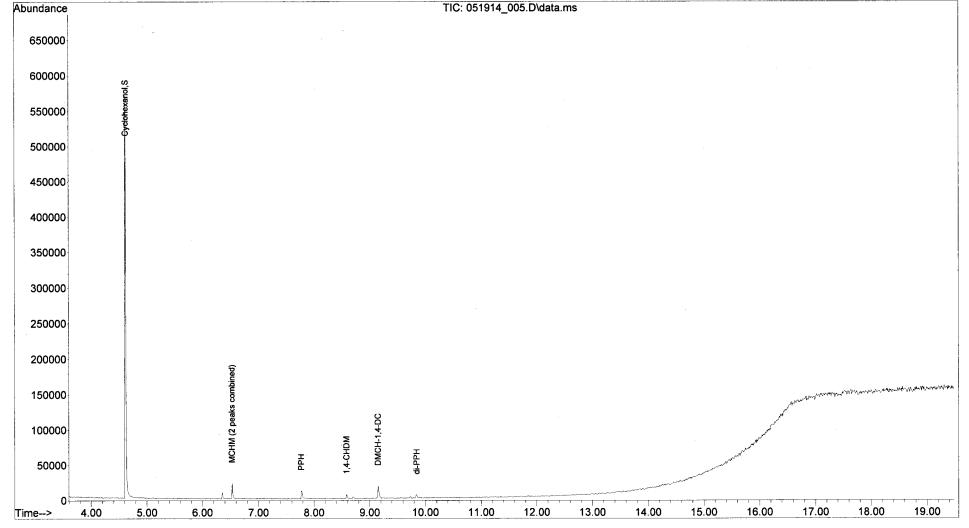
Misc : 3

ALS Vial : 5 Sample Multiplier: 1

Quant Time: Aug 06 11:42:27 2014

Quant Method: C:\msdchem\1\METHODS\ RP1581_051914.M

Quant Title : Freedom Industies (7)(C)
QLast Update : Wed Aug 06 11:32:39 2014



Data Path: C:\msdchem\1\DATA(b)(7)(C)(RP1581\

Data File : 051914_006.D

: 19 May 2014 2:32 pm Acq On

(b) (7) Operator Sample 5 ug/ml

: 5 Misc

ALS Vial : 6 Sample Multiplier: 1

RP1581 051914.M

Quant Time: Aug 06 11:43:33 2014
Quant Method: C:\msdchem\1\METHODS\
Quant Title: Freedom Industies
QLast Update: Wed Aug 06 11:32:39 2014
Response via: Initial Calibration

Compound	R.T.	QIon	Response	Conc Units De	ev(Min)
System Monitoring Compounds 1) Cyclohexanol	4.623	57	157595	48.86 ug/mL	0.01
Target Compounds 2) MCHM (2 peaks combined) 3) PPH 4) 1,4-CHDM 5) DMCH-1,4-DC 6) di-PPH	6.530 7.774 8.582 9.152 9.846	55 94 95 81 59	10861m 10474m 4360m 7673m 4799m	5.26 ug/mL 6.12 ug/mL 5.95 ug/mL 5.27 ug/mL 4.73 ug/mL	value

Data Path: C:\msdchem\1\DATA\b(0) (7)(C) RP1581\

Data File: 051914 006.D

Acq On : 19 May 2014 2:32 pm

Operator (b) (7)(C)
Sample : 5 ug/ml

Misc : 5

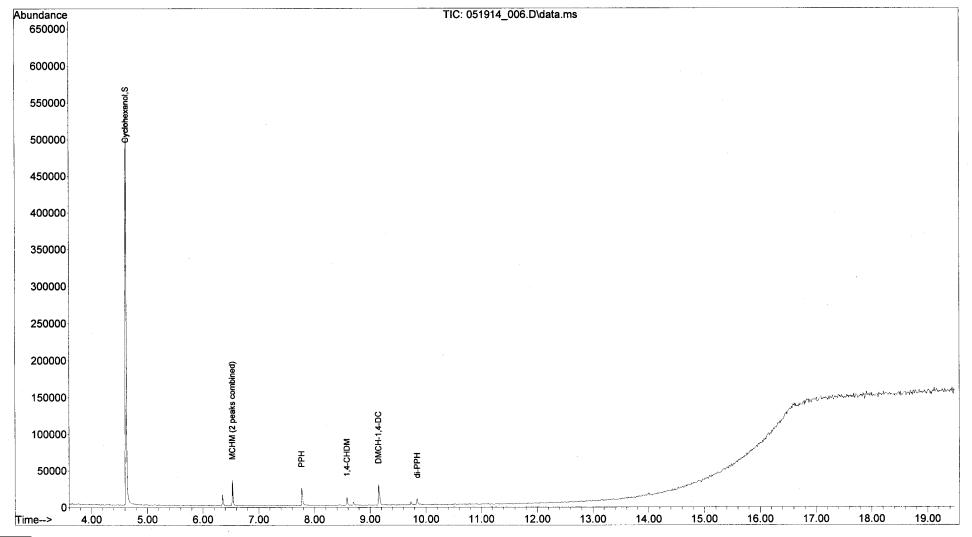
ALS Vial : 6 Sample Multiplier: 1

Quant Time: Aug 06 11:43:33 2014

Quant Method: C:\msdchem\1\METHODS\2010 (7) P1581_051914.M

Quant Title : Freedom Industies

QLast Update: Wed Aug 06 11:32:39 2014



Data Path: C:\msdchem\1\DATA(b)(7)(C) \RP1581\

Data File : 051914_007.D

: <u>19 M</u>ay 2014 Acq On 3:04 pm

(b) (7) Operator

: 10 ug/mLSample

: 10 Misc

ALS Vial Sample Multiplier: 1

Quant Time: Aug 06 11:44:19 2014

Quant Method: C:\msdchem\1\METHODS\
Quant Title: Freedom Industies
QLast Update: Wed Aug 06 11:32:39 2014
Response via: Initial Calibration RP1581 051914.M

Compound	R.T.	QIon	Response	Conc U	nits Dev	(Min)
System Monitoring Compounds 1) Cyclohexanol	4.623	57	151352	46.92	ug/mL	0.01
Target Compounds					Q7	alue
2) MCHM (2 peaks combined)	6.530	55	22258m	8.00	ug/mL	
3) PPH	7.774	94	21573	8.19	ug/mL#	55
4) 1,4-CHDM	8.582	95	8585m	7.90	ug/mL	
5) DMCH-1,4-DC	9.152	81	18423m	8.46	ug/mL	
6) di-PPH	9.846	59	11129m	7.55	ug/mL	

Data Path : C:\msdchem\1\DATA(0)(7)(C) RP1581\

Data File : 051914_007.D

Acq On : 19 May 2014 3:04 pm

Operator Sample

or (b) (7)(c) : 10 ug/mL

Misc : 10

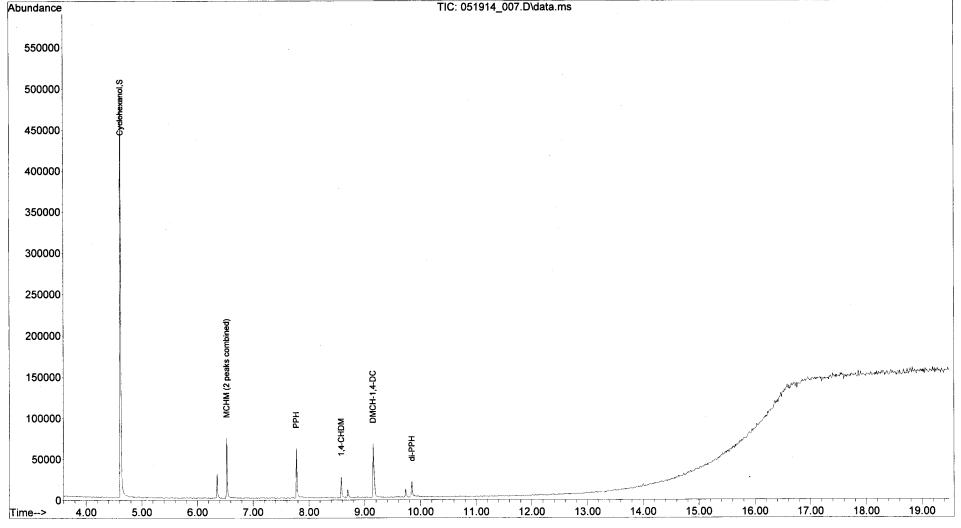
ALS Vial : 7 Sample Multiplier: 1

Quant Time: Aug 06 11:44:19 2014

Quant Method: C:\msdchem\1\METHODS(0)(7) RP1581_051914.M

Quant Title : Freedom Industies

QLast Update: Wed Aug 06 11:32:39 2014



Data Path : C:\msdchem\1\DATA\ (b) (7)(C) RP1581\

Data File : 051914_008.D

: 19 May 2014 : (b) (7) Acq On

Operator

Sample : 50 ug/mL

: 50 Misc

ALS Vial : 8 Sample Multiplier: 1

Quant Time: Aug 06 11:45:25 2014
Quant Method : C:\msdchem\1\METHODS\AMH_RP1581_051914.M
Quant Title : Freedom Industies
QLast Update : Wed Aug 06 11:32:39 2014
Response via : Initial Calibration

Compound	(b) (7)(C)		esponse	Conc Units Dev	(Min)
System Monitoring Compounds 1) Cyclohexanol	4.613	57	168333	52.19 ug/mL	0.00
Target Compounds				Qv	alue
MCHM (2 peaks combined)	6.530	55	163896m	41.96 ug/mL	
3) PPH	7.773	94	189499	39.50 ug/mL	94
4) 1,4-CHDM	8.582	95	74607m	38.26 ug/mL	
5) DMCH-1,4-DC	9.152	81	130770	41.79 ug/mL#	91
6) di-PPH	9.846	59	83192m	39.69 ug/mL	

Data Path : C:\msdchem\1\DATA (0) (7)(C) \RP1581\

Data File: 051914 008.D

Acq On : 19 May 2014 3:36 pm

Operator : (b) (7)(C)
Sample : 50 ug/mL

Misc : 50

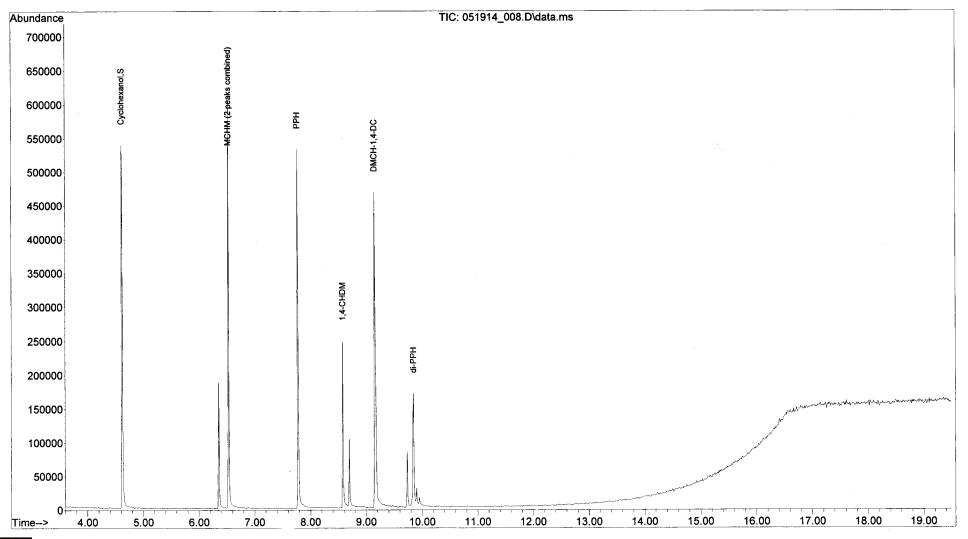
ALS Vial : 8 Sample Multiplier: 1

Quant Time: Aug 06 11:45:25 2014

Quant Method: C:\msdchem\1\METHODS (0) (7) RP1581_051914.M

Quant Title : Freedom Industies

QLast Update: Wed Aug 06 11:32:39 2014



Data Path : C:\msdchem\1\DATA\(\begin{array}{c} \begin{array}{c} \begin{ar

Data File : 051914_009.D

: 19 May 2014 : (b) (7)(C) Acq On

Operator Sample : 100 ug/mL

: 100 Misc

: 9 ALS Vial Sample Multiplier: 1

Quant Time: Aug 06 11:46:47 2014
Quant Method: C:\msdchem\1\METHODS\(\begin{array}{c} \begin{array}{c} (0) (7) \end{array} P1581_051914.M
Quant Title: Freedom Industies
QLast Update: Wed Aug 06 11:32:39 2014
Response via: Initial Calibration

Compound	R.T.	QIon	Response	Conc Units Dev	(Min)
System Monitoring Compounds 1) Cyclohexanol	4.623	57	167242	51.85 ug/mL	0.01
Target Compounds				Qv	alue
MCHM (2 peaks combined)	6.530	55	367546m	90.81 ug/mL	
3) PPH	7.774	94	441478	86.47 ug/mL	95
4) 1,4-CHDM	8.582	95	186886m	89.89 ug/mL	
5) DMCH-1,4-DC	9.152	81	293609	90.11 ug/mL#	91
6) di-PPH 	9.846	59 	214165m	98.11 ug/mL	

Data Path : C:\msdchem\1\DATA\(DATA\)

Data File : 051914_009.D

Acq On : 19 May 2014 4:09 pm

Operator : (b)

Sample : 100 ug/mL

Misc : 100

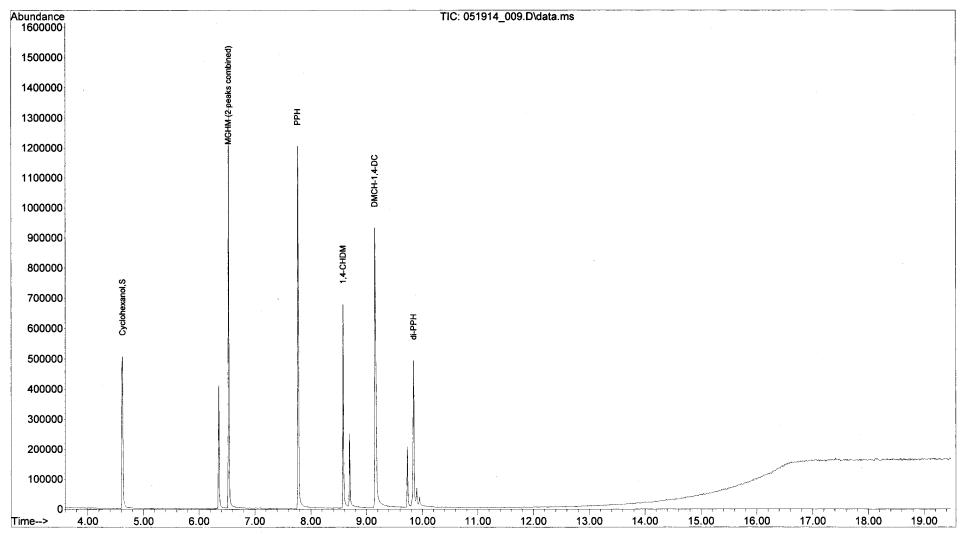
ALS Vial : 9 Sample Multiplier: 1

Quant Time: Aug 06 11:46:47 2014

Quant Method: C:\msdchem\1\METHODS\\(\begin{array}{c} \b

Quant Title : Freedom Industies

QLast Update: Wed Aug 06 11:32:39 2014



Data Path: C:\msdchem\1\DATA(b)(7)(C)\RP1581\

Data File : 051914_010.D

19 May 2014 (7)(C) Acq On 4:42 pm

Operator Sample

g/mL

Misc : 200

ALS Vial : 10 Sample Multiplier: 1

Quant Time: Aug 06 11:47:37 2014

Quant Method: C:\msdchem\1\METHODS
Quant Title: Freedom Industies
QLast Update: Wed Aug 06 11:32:39 2014
Response via: Initial Calibration RP1581 051914.M

Compound	(b) (7)(C)		Response	Conc Ur	nits Dev	(Min)
System Monitoring Compounds 1) Cyclohexanol	4.623	57	163415	50.66	ug/mL	0.01
Target Compounds					Qv	alue
MCHM (2 peaks combined)	6.530	55	789712m	192.05	ug/mL	
3) PPH	7.784	94	1023617	194.99	ug/mL	96
4) 1,4-CHDM	8.582	95	410254m	192.61	ug/mL	
5) DMCH-1,4-DC	9.162	81	639126	192.62	ug/mL#	92
6) di-PPH	9.846	59	527324m	237.79	ug/mL	•
			· 			

Data Path : C:\msdchem\1\DATA (b) (7)(C) RP1581\

Data File : 051914 010.D

Acq On : 19 May 2014 4:42 pm

Operator : (b)

Sample : 200 ug/mL

Misc : 200

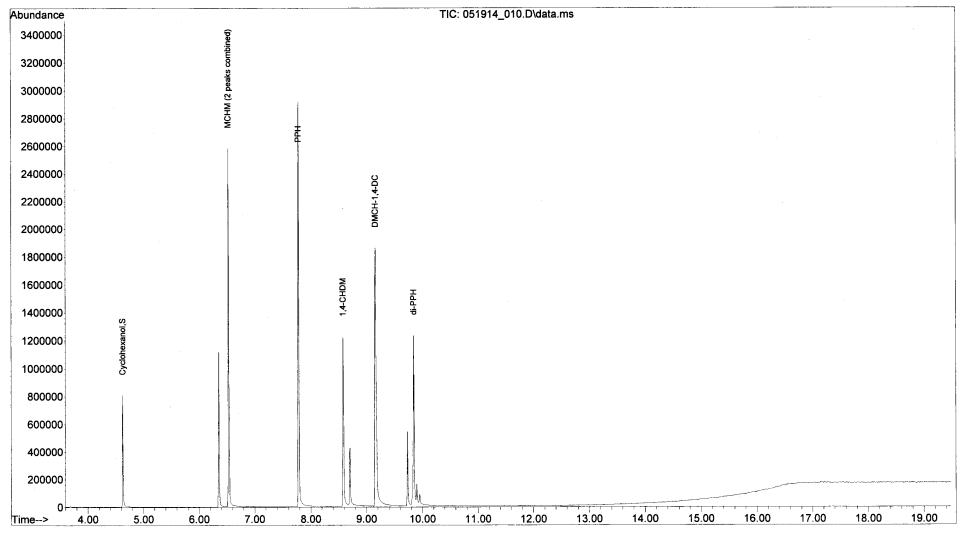
ALS Vial : 10 Sample Multiplier: 1

Quant Time: Aug 06 11:47:37 2014

Quant Method: C:\msdchem\1\METHODS (6) (7) RP1581_051914.M

Quant Title : Freedom Industies

QLast Update: Wed Aug 06 11:32:39 2014



METHOD CONTROL PARAMETERS ______

Method Information Method Sections To	For: C:\MSDCHEM\1\METHODS (C) RP1581.MRun:	1
	Method With Data ontrol Pre-Run Cmd/Macro =	

() Data Analysis Pre-Run Cmd/Macro = (X) Data Acquisition (X) Data Analysis () Instrument Control Post-Run Cmd/Macro = () Data Analysis Pre-Run Cmd/Macro = () Data Analysis Post-Run Cmd/Macro = () Data Analysis

Method Comments:

END OF METHOD CONTROL PARAMETERS

RP1581.M Thu Jun 05 14:28:37 2014

INSTRUMENT CONTROL PARAMETERS: Instrument 1

19.5 min

C:\MSDCHEM\1\METHODS (b) (7) RP1581.M Thu Jun 05 14:28:38 2014

Control Information

Sample Inlet : GC Injection Source : GC ALS Mass Spectrometer : Enabled

Oven

Run Time

Equilibration Time 0.5 min Oven Program On 35 °C for 2 min then 20 °C/min to 325 °C for 3 min

Front Injector

Syringe Size 10 µL Injection Volume 1 µL Injection Repetitions 1 Solvent A Washes (PreInj) 2 Solvent A Washes (PostInj) 3 8 µL Solvent A Volume Solvent B Washes (PreInj) 2 Solvent B Washes (PostInj) 3 . Solvent B Volume 8 µL Sample Washes 1 Sample Wash Volume 8 µL Sample Pumps
Dwell Time (PreInj)
Dwell Time (PostInj) 3 0 min 0 min 0 min 300 μL/min 6000 μL/min 300 μL/min 6000 μL/min 6000 μL/min Solvent Wash Draw Speed
Solvent Wash Dispense Speed Sample Wash Draw Speed
Sample Wash Dispense Speed
Injection Dispense Speed Viscosity Delay 0 sec Sample Depth Disabled

Back Injector

Front SS Inlet He Mode Split 275 °C Heater On 16.494 psi 47.305 mL/min Pressure On Total Flow On Septum Purge Flow On 3 mL/min Gas Saver On 30 mL/min After 2 min Split Ratio 20:1 Split Flow 42.195 mL/min

Back SS Inlet He

Mode Split
Heater Off
Pressure Off
Total Flow Off
Septum Purge Flow Off
Gas Saver Off
Split Ratio 100 :1

٦

Thermal Aux 2 {MSD Transfer Line}

Temperature Program 280 °C for 0 min On On

Run Time

19.5 min

Column #1

DB-5ms: 1653.60651

DB-5ms

325 °C: 30 m x 250 μ m x 0.25 μ m

In: Front SS Inlet He

Out: Vacuum

35 °C (Initial) 16.494 psi Pressure Flow 2.1098 mL/min52.535 cm/sec Average Velocity 0.95175 min Holdup Time Flow Program Off

2.1098 mL/min for 0 min

19.5 min Run Time

Front Detector µECD

300 °C On Heater Off Anode Flow

20 mL/min Makeup Flow On

Const Col + Makeup Off

Electrometer Off

Aux Pressure 1 He

Off Pressure Program

10 psi for 0 min 19.5 min Run Time

Aux Pressure 2 He

Off Pressure Program

10 psi for 0 min 19.5 min Run Time

Aux Pressure 3 He

Pressure Program Off 10 psi for 0 min

19.5 min Run Time

Signals Front Signal

Save Off Back Inlet Test Plot Save Off

Test Plot Save Off

MS ACQUISITION PARAMETERS

Save Off

General Information

Tune File : dftppnew051614.u

Acquistion Mode : Scan

Information _____

Solvent Delay : 3.50 min

EMV Mode : Relative Relative Voltage : -71 Resulting EM Voltage : 1812

[Scan Parameters]

Low Mass : 35.0 High Mass : 500.0

: 50 Threshold

A/D Samples 8

Sample #
Plot 2 low mass
Plot 2 high mass : 3 : 50.0 : 550.0

[MSZones]

MS Source : 230 C maximum 250 C : 150 C maximum 200 C MS Quad

END OF MS ACQUISITION PARAMETERS

TUNE PARAMETERS for SN: US73347311 ______

Trace Ion Detection is OFF.

EMISSION : 34.610 ENERGY : 69.922 REPELLER : 22.249 IONFOCUS : 90.157 ENTRANCE_LE : 0.000 EMVOLTS : 1882.353

Actual EMV : 1811.77 GAIN FACTOR : 2.25

AMUGAIN : 2075.000 AMUOFFSET : 126.750 FILAMENT : 1.000 1.000 :

DCPOLARITY: 0.000 ENTLENSOFFS: 17.318@ 3 17.318@ 50 15.310@ 69 13.302@131 13.804

0219 14.0550414 14.0550502 14.05501049

MASSGAIN : -453.000 MASSOFFSET : -39.000

END OF TUNE PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS

(b) (7) (C) RP1581.M

Percent Report Settings

Sort By: Signal

Output Destination

Screen: No Printer: No File: No

Integration Events: Meth Default

Generate Report During Run Method: Yes

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search Minimum Quality

C:\Database\NIST05a.L 0

Integration Events: Meth Default

Report Type: Summary

Output Destination

Screen: No Printer: No File: No

Generate Report During Run Method: Yes

Quantitative Report Settings

Report Type: Detailed

Output Destination

Screen: No Printer: No

File: epatemp.txt

Generate Report During Run Method: No

Freedom Industies

Calibration Last Updated: Tue Jun 03 14:32:34 2014

Reference Window: 2.00 Minutes Non-Reference Window: 1.00 Minutes

AMH_RP1581.M Thu Jun 05 14:28:37 2014

Page: 5

Correlation Window: 0.10 minutes Default Multiplier: 1.00 Default Sample Concentration: 0.00 Compound Information () 1) Cyclohexanol Ret. Time 4.613 min., Extract & Integrate from 4.113 to 5.113 min. Rel Resp. Pct. Unc. (rel) Signal Integration *** METH DEFAULT *** 57.10 Tqt *** METH DEFAULT *** Q1 82.10 57.10 20.0 *** METH DEFAULT *** 20.0 Q2 67.10 43.80 Lvl ID Conc (ug/mL) Response 50.000 1 156539 155813 50.000 2 3 50.000 169255 4 50.000 157595 151352 5 50.000 50.000 168333 6 50.000 7 167242 50.000 163415 Qualifier Peak Analysis ON Curve Fit: Avg. RF () 2) MCHM (2 peaks combined) Ret. Time 6.530 min., Extract & Integrate from 6.030 to 7.030 min. Rel Resp. Pct. Unc. (rel) Integration Signal Tgt 55.10 *** METH DEFAULT *** *** METH DEFAULT *** 39.40 20.0 97.10 Q1*** METH DEFAULT *** 81.10 30.30 Q2 20.0 Response Lvl ID Conc (ug/mL) 0.500 1.000 1464 3.000 3 4958 5.000 4 7581 5 15368 10.000 50.000 118295 6 265510 7 100.000 200.000 550216 Qualifier Peak Analysis ON Curve Fit: Linear 3) PPH () Ret. Time 7.784 min., Extract & Integrate from 7.284 to 8.284 min. Signal Rel Resp. Pct. Unc. (rel) Integration Tgt 94.10 *** METH DEFAULT *** 77.10 23.70 20.0 *** METH DEFAULT *** Q1 02 152.10 18.60 20.0 *** METH DEFAULT ***

2.0				
Lvl	ID	Conc (ug/mL)	Response
1		0.	500	548
2		1.	000	1506
3		3.	000	6257
4		5.	000	10053
5		10.	000	21573
6		50.	000	189499
7		100.	000	441478
8		200.	000	1023617

```
______
```

Curve Fit: Linear 4) 1,4-CHDM () Ret. Time 8.592 min., Extract & Integrate from 8.092 to 9.092 min. Rel Resp. Pct. Unc. (rel) Signal Integration Tgt 95.10 *** METH DEFAULT *** *** METH DEFAULT *** 67.10 41.40 20.0 01 Q2 41.10 53.20 20.0 *** METH DEFAULT *** Lvl ID Conc (ug/mL) Response 0.500 280 2 1.000 545 3 3.000 1859 5.000 3449 10.000 6555 50.000 58043 7 100.000 147139 200.000 315052 Qualifier Peak Analysis ON Curve Fit: Linear 5) DMCH-1,4-DC () Ret. Time 9.152 min., Extract & Integrate from 8.652 to 9.652 min. Signal Rel Resp. Pct. Unc.(rel) Integration Tgt 81.10 *** METH DEFAULT *** 108.10 20.0 *** METH DEFAULT *** 01 36.20 02 140.10 63.60 20.0 *** METH DEFAULT *** Lvl ID Conc (ug/mL) Response 0.500 2 1.000 1399 3 3.000 5113 4 5.000 7673 5 10.000 17864 50.000 6 130770 100.000 293609 200.000 639126 Qualifier Peak Analysis ON Curve Fit: Linear 6) di-PPH () Ret. Time 9.856 min., Extract & Integrate from 9.356 to 10.356 min. Signal Rel Resp. Pct. Unc. (rel) Integration Tgt 59.10 *** METH DEFAULT *** Q1 94.10 30.00 20.0 *** METH DEFAULT *** 210.20 9.00 20.0 *** METH DEFAULT *** 02 Lvl ID Conc (ug/mL) Response 0.500 221 726 2 1.000 3 3.000 2147 4 5.000 3145 5 10.000 7351 50.000 58083 7 100.000 154086 200.000 375689

Qualifier Peak Analysis ON Curve Fit: Linear

Qualifier Peak Analysis ON

END OF DATA ANALYSIS PARAMETERS

Thu Jun 05 14:28:39 2014